

STUDY OF THE LUBRICANT PROPERTIES OF
CARBON MONOFLUORIDE: $(CF_x)_n$

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(NASA-TM-75191) STUDY OF THE LUBRICANT
PROPERTIES OF CARBON NONFLUORIDE: (CF_{SUBx})
SUBn (National Aeronautics and Space
Administration) 18 p HC A02/MF A01 CSCL 11H

N78-16186

Unclas
G3/27 01897

Translation of "Étude des propriétés
lubrifiantes due monogluorure de graphite:
 $(CF_x)_n$ ", Colloques internationaux du
C.N.R.S., No. 233 - Polymères et lubri-
fication, pp. 441-450.



NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON, D. C.
DECEMBER 1977

1. Report No. NASA TM 75191	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle STUDY OF THE LUBRICANT PROPERTIES OF CARBON MONOFLUORIDE: $(CF_x)_n$		5. Report Date December 1977	
		6. Performing Organization Code	
7. Author(s) D. Play and M. Godet		8. Performing Organization Report No.	
		10. Work Unit No.	
9. Performing Organization Name and Address		11. Contract or Grant No.	
		13. Type of Report and Period Covered	
12. Sponsoring Agency Name and Address		14. Sponsoring Agency Code	
15. Supplementary Notes Translation of "Étude des propriétés lubrifiantes du monofluorure de graphite: $(CF_x)_n$ ", Colloques internationaux due D.N.R.S., No. 233 — Polymères et lubrification, pp. 441-450			
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17. Key Words (Selected by Author(s))		18. Distribution Statement Unlimited	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 19	22.

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D. Play and M. Godet*

ABSTRACT. Friction and wear tests were performed with rubbed carbon monofluorides derived from different types of carbon. The results are compared to those obtained under similar conditions with better known solid lubricants such as molybdenum disulfide (MoS_2). Best results were given by fluorated artificial graphite and coke. The coefficient of friction is practically independent of the fluorine-to-carbon ratio x where $0 < x < 1$, but wear decreases by a factor of 6 as x tends toward 1. For a range of variation of the severity factor PV expressed in $N/cm.sec$ (where P is the average pressure and V the velocity) contained between 10^3 and 2×10^5 , the wear measured with CF_x is significantly lower than that obtained with MoS_2 . A transfer film or third body which protects the surfaces is formed with CF_x . Beyond $400^\circ C$, thick and fragile oxide coatings are also formed.

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1. Introduction, Bibliography, and Definition of the Problem

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We know [1] that the design and construction of friction systems consists of solving simultaneously the problems of volume and the problems of surface. For the latter, in most cases, we have to consider the formation of a third body. Consequently, we have to know the conditions that should be satisfied, on the one hand, to assure proper operation (load, speed, temperature) and, on the other hand, to obtain minimum friction and sufficient lifetime, that is, acceptable friction or limited wear for a given length of time.

This problem of surface can be solved by the interposition of solid lubricants, among others, between the massive components. Among these, we can cite graphite, bisulfides of molybdenum and tungsten (MoS_2 , WS_2), the biselenides of molybdenum and tungsten (MoSe_2 and WSe_2), and organic compounds.

Since 1969, three different research teams have shown that graphite monofluoride $(\text{CF}_x)_n$ has solid lubricant characteristics.

Fusaro and Sliney [2, 3] indicate for a film applied "by rubbing" /44 that:

- graphite fluoride has good lubricant properties up to 400°C ;
- the friction coefficient is identical or superior to that of molybdenum or graphite bisulfide;
- the lifetime of the graphite fluoride film is at least six times greater than that of a molybdenum bisulfide film;
- humid air does not seem to cause change in the behavior of the graphite fluoride film;
- the graphite fluorination rate does not seem to affect the friction properties of the product for $0.7 < x < 1.12$.

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They add that, from the point of view of lifetime, graphite fluoride is preferable as an additive to molybdenum bisulphite for thin films of polyamide.

Gisser, Petronio, and Shapiro [4] show that:

- the lifetime of graphite fluoride films bonded with silicates or resins is 40 to 50% greater than that of graphite;
- 2% graphite fluoride in a lithium soap grease with a diester oil base performs satisfactorily up to 344° C, while the performance with the grease alone or with 2% graphite added is limited to 215° C.

Ishikawa [5] shows that the addition of graphite fluoride to greases and oils improves the lubricant properties of the compound. This is reflected in the increase of the load capacity, the reduction of the surface temperature, and the protection against jamming.

In conclusion, it seems that graphite fluoride $(CF_x)_n$ has interesting dry friction properties. However, there has not been a systematic study made of these products. Hence, the lubrication mechanism has remained unexplained until now. In the following, we propose to study the effect of the starting carbon, the fluorination rate x , the load, the velocity, and the temperature on the friction and the wear obtained with carbon fluorides, and to compare these values with those obtained with conventional solid lubricants.

This study will be performed on three different test machines.

2. Materials, Experimental Devices, Operating Modes

2.1. Materials and operating modes

The various products used in this study are shown in Table 1.

TABLE 1. MATERIALS

No.	Products	Source
1	CF _{x=0.96} ex. artificial graphite (GA)	P.C. U.K.
2	CF _{x=0.95} ex. natural graphite (GN)	
3	CF _{x=1.5} ex. activated charcoal	
4	CF _{x=1.3} ex. coke	
5	CF _{x=0} ex. GA	
6	CF _{x=0.45} ex. GA	
7	CF _{x=0.55} ex. GA	
8	CF _{x=0.60} ex. GA	
9	CF _{x=0.74} ex. GA	
10	CF _{x=0.87} ex. GA	
11	CF _{x=0.90} ex. GA	
12	CF _{x=1.1} ex. coke	Dow-Corning
13	MoS ₂ Molykote Z	
14	Mo Se ₂	Graphoil
15	W S ₂	
16	W Se ₂	

2.2. Experimental devices

Three machines have been used in the tests

2.2.a) A flat roller machine, described elsewhere [6]. The rapidity with which the grinding period (2 to 5 min) passes by enables us to set the total duration of the tests to one hour. The width of the wear marks (Figure 1) on the block is measured every 10 minutes after the first 5 minutes of the test, which corresponds to the accommodation of the surface. Since the width L of the track is constant for the entire length L of the impression, the wear can be evaluated by means of the transverse cross section S of this imprint, expressed in mm^2 . During the continuous movement the stops required to measure the width L do not affect the overall behavior as a function of time. The wear necessarily modifies the pressure distribution on the track. While the distribution is known at the beginning of the test and is given by the Hertz theory, it will not hold later, and so we define an average pressure P_{av} equal to the ratio of the

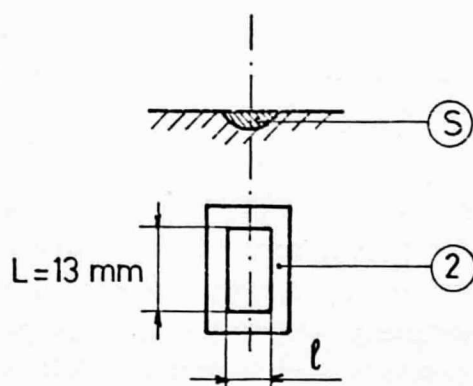


Figure 1. Wear track on the block

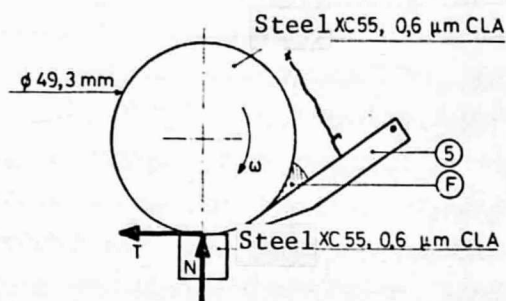


Figure 2. Diagram of the flat roller machine

load N normal to the surface of the wear track $l \times N$, so that $P_{av} = N/l \times L$.

The operating mode is identical for all the tests. The friction component (cup and block) is degreased with dimethylethylketone in an ultrasound bath. After drying in air, a simple application by rubbing by means of a lime spatula [5] enables us to coat the two friction surfaces. After mounting the test pieces on the machine, a reserve of lubricant (powder to be tested) is formed at F in Figure 2. The normal load N is applied when the rotational velocity of the axis is constant and the friction force T is recorded. The tests are carried out at normal ambience. /443

2.2.b) A scribe disk machine (Figure 3). A disk [1] made of XC38 steel is rotated very slowly ($\omega = 0.1$ radians/sec). A scribe [2] of the same material applies a constant load N by means of a support 3 and a lever 4. The friction force is recorded. The disk is heated by the Joule effect. The temperature, which is uniformly constant at the friction area, is measured by a thermocouple 6 placed at the center of the disk.

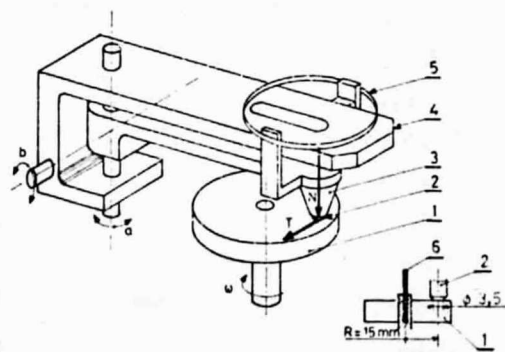


Figure 3. Diagram of the scribe disk machine

Before the friction test proper, lapping with diamond paste (Struers 7 μ) enables us to obtain good contact between the surfaces. After washing with trichloroethylene and degreasing with dimethylethylketone and ultrasound, the test pieces are mounted in their initial position. The powder to be tested is sprinkled on the disk. The test temperature and the constant rotation of the disk are set. The friction force T is recorded while the normal load N is applied. The tests are carried out at normal ambience.

2.2.c) An HTV machine, described elsewhere [7], simulates an axle/bushing contact (diameter 40 mm, length 40 mm). A test procedure identical to the above has been carried out.

2.2.d) The experimental analysis includes two sources of error: one due to the imprecision of the measurements, the other to the non-reproducibility of the phenomena:

· Errors in Measurement. The velocity is measured and kept within 1%. The error in the load can be as high as 0.5N. The duration of the test is defined within 30 sec. The boundaries of the wear groove are not defined rigorously, the wear profile varies slightly along the groove, the average width of the track is measured by means of a microscope, so that the accuracy is 5%.

· Errors due to the nonreproducibility of the phenomena. It is nearly impossible to obtain several identical tests for the same imposed conditions. Preliminary tests have enabled us to evaluate the reproducibility of the phenomena, when the loads, the velocities, and the times are controlled. We had to make two or three tests in each case.

3. Test Program

3.1. Study of the problem, tests on the flat roller machine

Variation of the friction coefficient in the wear S and the wear rate $\dot{S}$ have been obtained with materials 1 - 10 in Table 1, for a load of 90 N and a linear velocity at the contact $V = 5$ cm/sec.

3.2. Systematic survey: tests on the flat roller machine

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The results in the preceding tests have enabled us to select materials 11, 12, and 13 in Table 1 for the systematic study with three load conditions, 30, 90, 180 N, and three velocity conditions 5, 20, 80 cm/sec.

3.3. Lifetime of the lubricant film: flat roller and HTV machines

The changes in the friction coefficient are then determined with the transfer films already specified for materials 11 and 13 in Table 1, with a load and velocity condition of 90 N and 160 cm/sec. Changes in the friction coefficient have been determined for the same materials applied simply by rubbing prior to friction under load and velocity conditions of 3980 N and 2.9 cm/sec on the HTV machine.

3.4. Effect of temperature: disk scribe machine

Materials 11, 13, 14, 15, 16 in Table 1 enabled us to specify the changes in the friction coefficient as a function of temperature for a load of 10 N and a velocity of 0.15 cm/sec, and for temperatures between 20° C and 600° C.

4. Results and Discussion

4.1. Effect of the starting carbon material on the friction of the monofluoride $(CF_x)_n$ (test on the flat roller machine)

We can fluorate several types of carbon. We will seek to determine the effect of the starting carbon material (1 - 4 in Table 1) on the friction and the wear.

— The fluoride of artificial graphite $(CF_{x=0.96})_n$ (Figure 4a) has a friction coefficient of about 0.16, which increases with time.

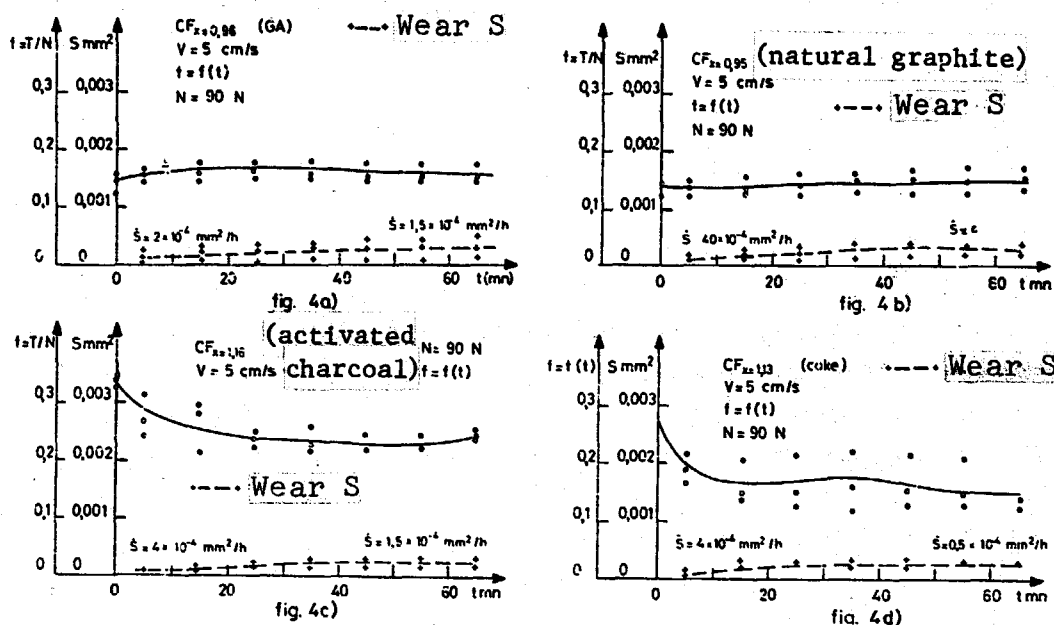


Figure 4. Behavior of the friction and the wear for various carbon fluorides

The wear, which increases rapidly at first, levels off in about an hour. The wear rate $\dot{S}$ reaches a value of $1.5 \cdot 10^{-4} \text{ mm}^2/\text{hr}$. A discontinuous film of 1 to 2/100 mm in thickness covers the friction surface. It is formed by a series of elongated fingers of about 1 to 2 mm in width (Figure 5).

— The fluoride of natural graphite ($\text{CF}_{x=0.95}$)_n (Figure 4b) has a nearly constant friction coefficient. A very low wear rate follows the considerable initial wear phase. The same transfer phenomenon appears on the friction ring.

— The fluoride of activated carbon ($\text{CF}_{x=1.16}$)_n (Figure 4c) has a high friction coefficient at the start of the test (approximately 0.34) which later decreases but remains about 0.2. High at the start, the wear rate levels off at the end of the test at $\dot{S} = 1.5 \times 10^{-4} \text{ mm}^2/\text{hr}$. The transfer to the test ring is practically non-existent and the wear track has tears in the material identical to those of jamming.

— The friction coefficient of coke fluoride ($\text{CF}_{x=1.13}$)_n (Figure 4d) goes from a high value (about 0.3) to an average value

at the end of the test (about 0.15). The wear rate becomes very low at the end of the test ($\dot{S} = 0.5 \cdot 10^{-4} \text{ mm}^2/\text{hr}$). The transfer film is thin and uniform. Some tearing points appear on the friction track.

These first tests (Table 2) show that:

TABLE 2. EFFECT OF INITIAL CARBON ON THE FRICTION OF FLUORIDES

$V = 5 \text{ cm/s}$ $P_{av} = 20 \text{ N/mm}^2$	$CF_x = 0.96$ (GA)	$CF_x = 0.95$ (GN)	$CF_x = 1.15$ (activated charcoal)	$CF_x = 1.13$ (coke)
f	0,155	0,135	0,27	-
$\dot{S} \cdot 10^4 \text{ mm}^2/\text{h}$	2.	4.	4.	7.



Figure 5. Ring after friction

— the friction coefficient obtained with natural graphite monofluoride is the lowest. On the other hand, artificial graphite monofluoride gives the lowest rate;

— the wear rates of the monofluorides of natural graphite ($CF_x = 0.95$)_n and of activated carbon ($CF_x = 1.16$)_n are similar;

— the wear is low when transfer films are formed.

In some, the leafy structure of the fluorated graphites seems to give the lowest values of the friction coefficient. In the case of coke monofluoride ($CF_x = 1.13$)_n the wear is aggravated by the presence of coke which, since it is not a solid lubricant, attacks the surface by abrasion. In conclusion, the resistance to wear of the natural and artificial graphite monofluorides are essentially identical and they are better than that of coke and activated charcoal monofluorides.

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4.2. Effect of the fluorination rate x on artificial graphite (test on the flat roller machine for materials 11, 12, 13, in Table 1)

Figure 6 shows the variation of the friction and the wear rate as a function of fluorination rate. The friction coefficient increases slightly up to 0.18 for values of x close to 1. On the other hand,

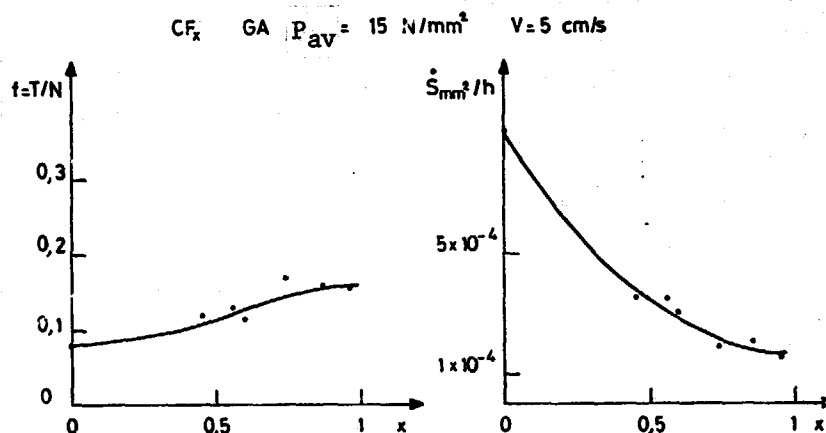


Figure 6. Effect of the fluorination rate

the wear rate $\dot{S}$ decreases from 6 to 1 when x increases from 0 to 1. The artificial graphite monofluorides $(CF_x)_n$ with $x = 0.45, 0.56, 0.60, 0.74, 0.87$, and 0.96 , form transfer film on the friction ring (Figure 5). Therefore the changes in the fluorination rate affect essentially the wear rate, that is, the degree of protection of the surfaces, and the likelihood of forming a third intermediate body.

4.3. Systematic study of the changes in load and velocity conditions (flat roller machine)

Tables 3, 4, and 5 summarize the results of these tests. The trends in the friction and wear curves are identical to those in Figure 4. The transfer film obtained with artificial graphite monofluoride is always discontinuous. Analysis of this transfer film by x-ray diffraction (Debye-Sherrer method) shows the presence of oriented graphite. Furthermore, mass spectroscopic analysis of

TABLE 3

$P_{av} = 600 \text{ N/cm}^2$		$(CF_{x=0,9})_n$ GA	$(CF_{x=1,1})_n$ coke	$M_o S_2$ (M)
$V = 5 \text{ cm/s}$ $P_{av} V = 3000$	f_{moy} $\dot{S} \times 10^4$	0,11 1	0,29 1,8	0,10 10
$V = 20 \text{ cm/s}$ $P_{av} V = 12000$	f_{moy} $\dot{S} \times 10^4$	0,14 2	0,25 5	0,12 11
$V = 80 \text{ cm/s}$ $P_{av} V = 48000$	f_{moy} $\dot{S} \times 10^4$	0,17 1	0,15 11	0,11 28

TABLE 4

$P_{av} = 1200 \text{ N/cm}^2$		$(CF_{x=0,9})_n$ (GA)	$(CF_{x=1,1})_n$ (coke)	$M_o S_2$ (M)
$V = 5 \text{ cm/s}$ $P_{av} V = 6000$	f_{moy} $\dot{S} \times 10^4$	0,13 4	0,22 2	0,15 13
$V = 20 \text{ cm/s}$ $P_{av} V = 24000$	f_{moy} $\dot{S} \times 10^4$	0,16 3	0,2 4	0,12 15
$V = 80 \text{ cm/s}$ $P_{av} V = 96000$	f_{moy} $\dot{S} \times 10^4$	0,17 4,8	0,13 8,8	0,07 10

these particles at ambient temperature shows the presence of fluorine F, of hydrogen fluoride HF, and of decomposition products CF_2 , CF_3 , CF_4 .

The effect of pressure on the friction coefficient of graphite monofluoride is small for all expected velocities.

The wear rate $\dot{S}$ increases with the load. The wear rate at 5 cm/sec increases from 1 to 20 with the load, while at 20 cm/sec it goes from 2 to 4. On the other hand, for a pressure of 2400 N/cm² and an increase in velocity from 5 to 20 cm/sec, the wear rate $\dot{S}$ decreases from 5 to 1. For the same pressure, a velocity of 80 cm/sec represents a thermal environment unfavorable for maintaining the transfer film. The protection becomes less efficient; the wear rate increases. Destruction sets in for more severe velocity conditions (see paragraph "lifetime of the film").

TABLE 5

$P_{av} = 2400 \text{ N/cm}^2$		$(CF_{x=0,9})_n$ (GA)	$(CF_{x=1,1})_n$ (coke)	$M_o S_2$ (M)
$V = 5 \text{ cm/s}$ $P_{av} V = 12000$	f_{moy} $\dot{S} \times 10^4$	0,13 21	0,18 6	0,13 10
$V = 20 \text{ cm/s}$ $P_{av} V = 48000$	f_{moy} $\dot{S} \times 10^4$	0,16 4	0,15 10	0,1 32
$V = 80 \text{ cm/s}$ $P_{av} V = 192000$	f_{moy} $\dot{S} \times 10^4$	0,14 14	0,13 30	0,09 15

The friction coefficient of coke monofluoride decreases for increasing pressures. At a slipping velocity of 5 cm/sec, the wear rate is nearly constant for the three pressures. For velocities of 20 and 80 cm/sec, the wear rate increases with contact pressure.

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For comparison, we note that molybdenum bisulfide has friction coefficients that decrease slightly as the pressure and the velocity increase. The wear rate is relatively high for velocities of 20 cm/sec. To compare the various values of wear rate, we will use, for lack of more significant criteria, the parameter $p_{av}V$ currently used in practice. It is a severity criterion by means of a certain representation of the thermal conditions at the contact.

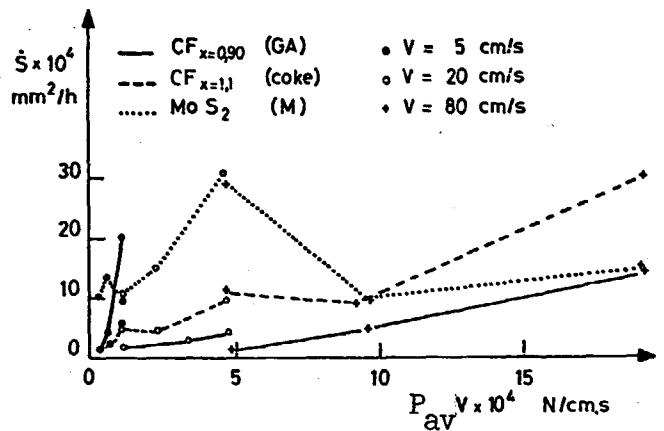


Figure 7. Variation of the wear rate as a function of the parameter $p_{av}V$

Relative to molybdenum bisulfide (MoS_2), we find that:

— the protection of the surface is better with artificial graphite fluoride ($CF_{x=0.9}$)_n up to values of pV equal to $2 \cdot 10^5$;

— protection with coke fluoride ($CF_{x=1.1}$)_n is limited for values of $pV - 10^5$.

4.4. Lifetime of the transfer films for materials 11 and 13 in Table 1

All the carbon fluorides like molybdenum bisulfide protect the surfaces when they are likely to form a transfer film. In the previous tests, the contact was supplied with lubricant continuously. Because of this, we were not able to evaluate the resistance of these transfer films as a function of time, that is, their lifetime. It is important to know the magnitude of the duration of protection provided by the films obtained from artificial graphite fluoride and molybdenum bisulfide. We will determine this for two extreme conditions of operation. After forming a film for fifteen minutes of operation

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on the flat roller machine, the supply of lubricant (Figure 2f) is removed.

The results in Figure 8a and b show that the lifetime of the transfer films is not negligible and varies between 30 minutes and 3 hours. For graphite fluoride, we note that a thin and uniform

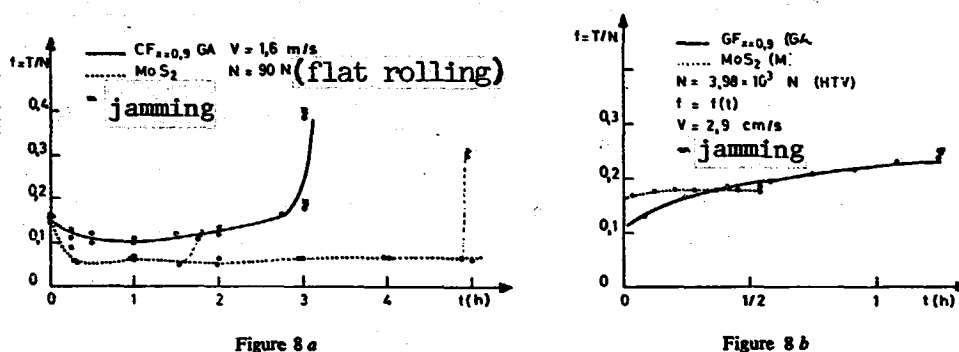


Figure 8. Lifetime of the transfer film

transfer film covers the friction ring at the start of the test. Before jamming, the friction coefficient increases gradually, then falls back to its constant value (behavior of friction in cracks). There is successive elimination and re-formation of the film. When jamming sets in, the film is destroyed completely.

4.5. Effect of temperature on the friction coefficient (tests on the scribe disk machine for materials 11, 13, 14, 15, 16, in Table 1)

Two types of tests have been carried out, some at constant temperature, others at increasing temperature. For the same value of temperature, the friction coefficients are identical in the two cases.

Artificial graphite monofluoride has a friction coefficient which decreases as a function of temperature (Figure 9). From 200 to 300° C, the friction coefficient F decreases slowly and reaches a minimum at 460° C, which is followed by a drastic increase. The analysis of the starting material in a Uguine-Eyraud B60 thermal balance in ambient air shows that the decomposition of the product

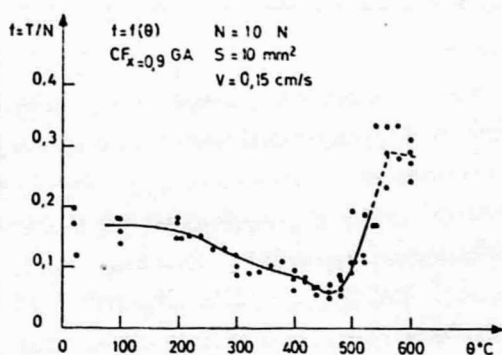


Figure 9. Effect of temperature on the friction of graphite fluoride

becomes appreciable toward 390° C.

A very rapid reaction sets in at 500° C, with considerable liberation of fluorine and splatter of graphite. Between 200° C and

300° C, a film of the same appearance as that found on the rings (Figure 5) is formed on the friction track of the disk. The test at 400° C shows the appearance of a slightly granulous track, as the oxide layer does not detach from the surface. On the other hand, tests at 500 and 600° C (Figure 10) show a very fragile 0.4 mm layer of Fe_2O_3 .



Figure 10. Disk after friction at 500° C

For temperatures above 450° C, the compound FeF_3 , likely to be formed, gives rise to Fe_2O_3 with liberation of fluorine and hydrogen by reacting with the oxygen in the air. These reaction products pass through the porous oxide layer. Thus this process leads to a series of reactions that are stabilized, of course, when the oxide layer becomes very thick. Tests carried out with Z100C17 samples (martensitic stainless steel, corresponding to 440° C) with a hardness of 60RHc also produce an oxide layer of 0.2 mm.

The products being compared (MoS_2 , MoSe_2 , WS_2 , WSe_2) have values of friction that decrease slowly to a minimum at 300° C and which increase considerably for higher temperatures (Figure 11). In conclusion, it seems that:

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— artificial graphite monofluoride retains its lubricant properties up to 400° C;

— its use is limited for higher values.

It is an advantageous solid lubricant for operating temperatures between 100 and 400° C.

5. Conclusion

This study has enabled us to reach conclusions already proposed in the literature concerning particularly:

- the favorable lubricant properties up to 400° C;
- a friction coefficient identical to or better than that of molybdenum disulfide,
- under favorable conditions, a wear rate with CF_x film at least 6 times smaller than that obtained with MoS_2 .

This study enables us to add the following conclusions:

- 1) The fluorination of artificial graphite and sometime even that of coke is advantageous for friction applications.
- 2) The increase in the fluorination of graphite (GA) barely affects the friction coefficient, but considerably decreases the wear rate.
- 3) The protection is related to the formation of a film on the surface created by an in situ chemical transformation of the starting material.

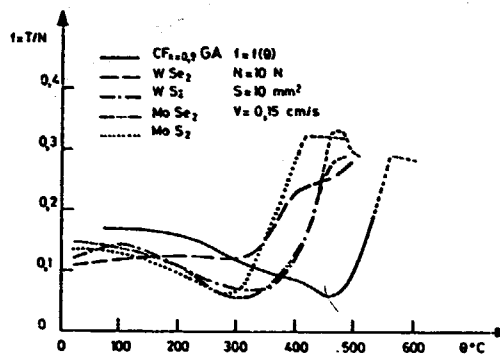


Figure 11. Effect of temperature on the friction of various lubricants

4) The use of CF_x for friction above $400^\circ C$ leads to the formation of thick and fragile oxide layers.

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This study has shown that graphite fluoride applied by "rubbing" is very promising. Its industrial use not requires the study of methods of applying it before forming the third intermediate body before or during the friction operation.

6. Acknowledgements

This research was financed by a grant from the Ugine-Kuhlmann Chemical Products Company to the Contact Mechanics Laboratory of the INSA (National Institute of Applied Sciences at Lyon).

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